PATENT ABSTRACTS OF JAPAN

(11)Publication number:

2000-344973

(43)Date of publication of application: 12.12.2000

(51)Int.Cl.

CO8L 23/10

COSK 5/02

CO8K 5/13

CO8K 5/57

(21)Application number: 11-157381

157381 (71)Applic

(71)Applicant: FUKUYU KAGAKU YUGENKOSHI

(22)Date of filing:

04.06.1999

(72)Inventor: RYO SHORIN

(54) FLAME RETARDANT COMPOSITION FOR POLYPROPYLENE

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a flame retardant composition imparting flame retardance or fire resistance to articles made of polypropylene when mixed into a polypropylene resin.

SOLUTION: This composition is obtained by compounding 20-80 wt.% hexabromocyclododecane 1-70 wt.% halobisphenol composition, i.e., tetrabromobisphenol A-bis(2,3-dibromopropyl ether) or tetrahalobisphenol A, 1-60 wt.% halodiphenyl eter i.e., octabromodiphenyl oxide or decabromodiphenyl oxide and 1-10 wt.% thermal stabilizer.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

* NOTICES *.

Japan Patent Office is not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2. **** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] 20 to 80% of the weight of a hexa BUROMOSAI clo dodecane (Hexabromocyclododecane;HBCD), 1 to 70 made into a tetrabromobisphenol A-screw (2, 3-dibromopropyl ether) (TBBPA-DBP) or tetrapod bisphenol A% of the weight of a halogen bisphenol compound, Octave ROMOJI phenyl 1 to 60 made into oxide (Octabromodiphenyloxide) or deca BUROMO diphenyloxide (Decabromodiphenyl oxide)% of the weight of a halogen diphenyl ether, The flame-retarder constituent of polypropylene characterized by being formed by 1 to 10% of the weight of the thermostabilizer, and the above.

[Claim 2] The flame-retarder constituent of polypropylene according to claim 1 characterized by making the aforementioned thermostabilizer into an organic tin stabilizer.

[Claim 3] 20 to 80% of the weight of a hexa BUROMOSAI clo dodecane (Hexabromocyclododecane;HBCD), A screw (3) [5-dibromo-4] A phenyl sulfone (2, 3-dibromo pro PUKISHI) 1 to 50 made into sulfone or tris (2, 3-dibromopropyl) isocyanurate (Tris(2, 3-dibromopropyl) isocyanurate)% of the weight of a compound, (Bis(3, 5-dibromopropxy) phenyl) Octave ROMOJI phenyl 1 to 60 made into oxide (Octabromodiphenyloxide) or deca BUROMO diphenyloxide (Decabromodiphenyl oxide)% of the weight of a halogen diphenyl ether, The flame-retarder constituent of polypropylene characterized by being formed by 1 to 10% of the weight of the thermostabilizer, and the above.

[Translation done.]

* NOTICES * ·

Japan Patent Office is not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2. **** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention] [0001]

[The technical field to which invention belongs] this invention relates to the flame-retarder constituent of a kind of polypropylene, and relates to the flame-retarder constituent which gives good fire retardancy or good refractoriness to the goods which mixed into polypropylene resin and were manufactured by polypropylene resin.

[Description of the Prior Art] It is light, a shock is borne, and it is equal to bending, and is glossy, the electric property has the outstanding point of being good, and polypropylene resin is applied into various kinds of large molding materials. However, since polypropylene resin belongs to a polyolefine compound, itself tends to burn. For this reason, a limit occurs in the direction of application. For this reason, addition of the flame retarder to polypropylene resin is very effective when extending the application range. It is very high and the fireproof demand of the polypropylene resin used for an electric supply is already UL-94 to the 1998 o'clock. It is obliged to attain the fireproof level of V-0. [0003] In the commercial scene, the bromide (bromide) is used as a flame retarder of polypropylene resin. In the flame retarder generally used, it is deca BUROMO diphenyloxide (Decabromodiphenyl oxide). Tetrabromobisphenol A (Tetrabromobisphenol A), A tetrabromobisphenol A-screw (2, 3-dibromopropyl ether) (Tetrabromobisphenol A-bis(2, 3-dibromoprophlether); TBBPA-DBP), A hexa BUROMOSAI clo dodecane (Hexabromocyclododecane), Octave ROMOJI phenyl Oxide (Octabromodiphenyl oxide), The screw (3, 5-dibromo -4 (2, 3-dibromo pro PUKISHI) phenyl) sulfone sulfone (Bis(3, 5-dibromopropxy) phenyl) And there are products, such as tris (2, 3-dibromopropyl) isocyanurate (Tris(2, 3-dibromopropyl) isocyanurate). Usually, the bromide of a single kind is used as a flame retarder, and, for this reason, the greatest effect cannot be attained. The fire-resistant effect over the polypropylene resin of the bromide flame retarder of the common knowledge to the following is examined.

- 1. It is an effective fire-resistant additive, the addition to polypropylene resin is 12%, and a hexa BUROMOSAI clo dodecane (Hexabromocyclododecane;HBCD) is 6% of Sb 2O3. When blended, it is UL-94 to polypropylene resin. It is made to attain the fireproof level of V-0. However, the HBCD itself was an aliphatic bromide, it was poor, and decomposed immediately at the time of processing fabrication of a resin, and heat-resistant stability brought about discoloration of a moldings, and destruction of a processing facility. For this reason, the fire-resistant effect in an experiment has not been attained in actual operation.
- 2. Deca BUROMO diphenyloxide (Decabromodiphenyloxide; OCTA) is made into a high bromine content, bears pyrolysis, and is the bromide object fire retardancy additive of a high-melting point. However, it cannot decompose, when polypropylene resin burns, since the pyrolysis temperature is too high, and a lot of bromines cannot be generated, but it is [this] a sake. It is low, when used for polypropylene resin (94 V-UL-0 level), it is used at no less than 25% of a high rate, and the fire-resistant effect is 8% of Sb 2O3. It was blended, and such a high additive content formed the frost phenomenon, and the shock resistance, the bending strength, and the glossiness of polypropylene resin were affected.
- 3. Octave ROMOJI phenyl Although it considers as a high bromine content like DECA, oxide (Octabromodiphenyl oxide) has the low melting point comparatively with low pyrolysis temperature, and when applied to polypropylene resin, it has a comparatively good fire-resistant effect.
- 4. TBBPA-DBP is an effective fire-resistant additive in a commercial scene, and the addition is Sb 2O3 of) and the 4-6 section to (polypropylene resin at the ten to 17 section. It blends and is UL-94. The fireproof demand of V-0 is attained. However, the compatibility of it and polypropylene resin was poor, even if it reduced the amount used even in the three sections, the frost phenomenon was generated, and the price was high, and the manufacturing cost of the polypropylene resin product containing it became high.

- 5. Tetrabromobisphenol A (Tetrabromobisphenol A, TBBPA) is a cheap bromide and has a thermolysis curve similar to TBBPA-DBP. However, it has a low bromine content and the comparatively high melting point comparatively. It is UL-94, when it is single and is used for polypropylene resin. Although the demand of V-2 can be attained easily, it is UL-94 by the relation of compatibility. The demand of V-0 was not able to be attained.
- 6. The screw (3, 5-dibromo -4 (2, 3-dibromo pro PUKISHI) phenyl) sulfone (Bis(3, 5-dibromopropxy) phenyl) sulfone is an effective fire-resistant additive to polypropylene resin. When added by polypropylene resin, a frost phenomenon is not generated, but it is 5% of Sb 2O3 in 10% of additions. It is UL-94 by being used blending. V-0 One eighth of demands are attained. However, for the low reason, the melting point brought about the problem on processing very much, and its price was very high, it raised the manufacturing cost of a polypropylene resin product, and was not economical.
- 7. If tris (2, 3-dibromopropyl) isocyanurate (Tris(2, 3-dibromopropyl) isocyanurate; TAUC-6B) contacts the skin, it will do harm. in China, it is produced and used, the fireproof effect over the polypropylene resin is good, and the amount used may be 10% -- having -- 5% of Sb 2O3 it blends -- having -- UL-94 V-0 One eighth of fireproof demands are attained. However, control of thermal stability was difficult, discoloration occurred for the product, the fire prevention effect was unstable and there was a fault, such as doing damage to a processing facility.

[Problem(s) to be Solved by the Invention] It is making into the technical problem for this invention to offer a kind of flame-retarder constituent which was made based on the detailed research on the combustion characteristics of polypropylene resin, and the mechanism of the combustion prevention to the polypropylene resin of various bromides, and analysis, sets up the compounding ratio of the bromide which changes suitably with various kinds of fire-resistant properties and reaction mechanisms in each stage of polypropylene resin combustion of a bromide, is effective and has economic effects by still more exact experiment.

[0005]

[Means for Solving the Problem] Invention of a claim 1 20 to 80% of the weight of a hexa BUROMOSAI clo dodecane (Hexabromocyclododecane;HBCD), A tetrabromobisphenol A-screw (2) [3-dibromopropyl] 1 to 70 made into the ether (Tetrabromobisphenol A-bis(2, 3-dibromoprophl ether);TBBPA-DBP) or tetrapod bisphenol A% of the weight of a halogen bisphenol compound, Octave ROMOJI phenyl 1 to 60 made into oxide (Octabromodiphenyloxide) or deca BUROMO diphenyloxide (Decabromodiphenyl oxide)% of the weight of a halogen diphenyl ether, It is considering as the flame-retarder constituent of polypropylene characterized by being formed by 1 to 10% of the weight of the thermostabilizer, and the above. Invention of a claim 2 is taken as the flame-retarder constituent of polypropylene according to claim 1 characterized by making the aforementioned thermostabilizer into an organic tin stabilizer. Invention of a claim 3 20 to 80% of the weight of a hexa BUROMOSAI clo dodecane (Hexabromocyclododecane;HBCD), A screw (3) [5-dibromo-4] A phenyl sulfone (2, 3-dibromo pro PUKISHI) 1 to 50 made into sulfone or tris (2, 3-dibromopropyl) isocyanurate (Tris(2, 3-dibromopropyl) isocyanurate)% of the weight of

a compound, (Bis(3, 5-dibromopropxy) phenyl) Octave ROMOJI phenyl 1 to 60 made into oxide (Octabromodiphenyloxide) or deca BUROMO diphenyloxide (Decabromodiphenyl oxide)% of the weight of a halogen diphenyl ether, It is considering as the flame-retarder constituent of polypropylene characterized by being formed by 1 to 10% of the weight of the thermostabilizer, and the above. [0006]

[Embodiments of the Invention] The flame-retarder constituent of the polypropylene of this invention includes the

following. namely, -- although 1. aliphatic bromide, a hexa BUROMOSAI clo dodecane (Hexabromocyclododecane;HBCD), and this have a high bromine content, a low decomposition temperature, and the high fire retardancy effect and polypropylene resin can be made to attain a considerable fire-resistant high demand by the low heatproof decomposition temperature at the time of independent use -- however, the glossiness of a resin -- destroying -- a product -- etiolation -- or carry out melanism, and a work machine is made to generate corrosion, and big loss is also hung down This phenomenon is improvable by adding an effective thermostabilizer, therefore in this invention, it is used as an inhibitor of a low-temperature flame, the temperature of a combustion object is lowered, and continuation combustion of a flame is prevented. It is desirably made into 20 to 50 % of the weight, the rate of a use rate is 20 to 80 % of the weight, since it can offer a lot of effective bromines at the time of low temperature, it lowers combustion temperature, and it attains the purpose which erases flame.

2. A tetrabromobisphenol A-screw (2, 3-dibromopropyl ether) (Tetrabromobisphenol A-bis(2, 3-dibromoprophl ether); TBBPA-DBP) has the feature of the decomposition-temperature curve and the fire-resistant high efficiency of a high bromine content and the degree of inside, and let it be an effective fire-resistant additive to polypropylene. The

decomposition curve engages with the combustion characteristics of polypropylene resin comparatively. However, the compatibility with polypropylene resin of the molecular structure is poor, it generates a frost phenomenon easily, and brings about ***** in **, and is the process of production of a tetrabromobisphenol A-screw (2, 3-dibromopropyl ether). The reaction itself is disadvantageous for control of generation conditions, purification of a product is difficult, purity runs short and the thermal stability can be strengthened with using the suiting binder and thermostabilizer. Since manufacturing process control is [that the fire-resistant effect can be strengthened] difficult, Though a unit price is not high, purity is insufficient, and to addition to polypropylene resin, it is incongruent. However, a good product offers a good fire-resistant effect at the time of polypropylene resin combustion, therefore is adopted during combination of this invention, offers a suitable fire-resistant effect, and raises fire-resistant efficiency. The rate of a use rate is desirably made into 25 to 70% 70% from 1, offers a proper decomposition bromide at the temperature to which polypropylene resin burns, suppresses the self-piloting ignition of a flame, and attains a fire-resistant effect. This product offers the product which can substitute for other, a part, or the fire-resistant effect which this tetrabromobisphenol A has a temperature part integral curve near a tetrabromobisphenol-A-screw (2, 3-dibromopropyl ether) by the ability being able to substitute for all, for example, being able to substitute tetrabromobisphenol A (Tetrabromobisphenol A:TBBPA), has a low bromine content, has some low fire-retardancy effects, and is equivalent to a tetrabromobisphenol-A-screw, and cannot generate a frost phenomenon comparatively easily.

- 3. Deca BUROMO Diphenyloxide (DecabromodiphenyloxIde) Are the bromide fire retardancy additive of a high bromine content, dissociation temperature, and a high-melting point, polypropylene resin burns continuously, and when temperature is high, it decomposes. Emit a decomposition product, make temperature of combustion of a resin low, and a fire-resistant effect is attained. bromination -- And if a lot of deca BUROMO diphenyloxide is used, polypropylene resin will not form **** at the time of combustion, but a plug operation is demonstrated (capacity is higher than 25 % of the weight), and the rate of a use rate in the inside of this invention is made into 1 to 60%, and is desirably made into 10 to 40%. This is an octave ROMOJI phenyl. Oxide (Octabromodiphenyloxide) can be substituted and it is this octave ROMOJI phenyl. Although oxide has comparatively high thermal stability like deca BUROMO diphenyloxide, it has low pyrolysis temperature comparatively, has the comparatively suitable melting point, fuses it completely in the addition process to polypropylene resin, attains a comparatively good dispersion effect, and demonstrates a comparatively good fire-resistant effect.
- 4. A thermostabilizer and application of this thermostabilizer absorb the bromine which a flame retarder generates in early decomposition, it prevents that it serves as a catalyst of the bromide which is not disassembled [other], the resolving time of a bromide is delayed, and, generally it is used as a thermostabilizer of a polyvinyl chloride. However, since it is made into organic-metal salts, an excessive thermostabilizer brings about the fall of a fire-resistant effect, for this reason, an effect chooses a comparatively good thermostabilizer, and reduces the amount used, enables it to obtain a good heat stabilizing effect, and prevents the fall of a fire-resistant effect. It is good that a tin content uses 32% or more of tin stabilizer, and the amount used is desirably taken as 2-70% 10% from 1.

[0007] It turns out that this invention is the effective flame-retarder constituent which fitted addition into polypropylene resin by experiment. The experiment used the 2 shaft EKUSUTO loader, by distribution of the following [160 degrees C], it mixed, and it kneaded it, and was rolled out with the piece machine of **, manufactured 0.5mm in thickness, and the 1.0mm piece of a sample, and performed the fire-resistant effect examination. The result was following table 1 passage.

[Table 1] ポリプロビレン 樹脂	防火剤(%)	Sb:0;(%)	分散助剤(%)	#UL94 V2/V0
87. 5	7. 986	3, 514	1	1mm V-0 0.5mm V-2
85. 714	9. 388	3, 755	1.143	1mm Y-0 0.5mm Y-2
83, 333	10. 952	4. 381	1. 334	1mm V-0 0.5mm V-2

[0008] Moreover, the actual operation experiment at the time of ordinary users' operation process was conducted at 200 degrees C using the 2 shaft EKUSUTO loader. The composition and a result are as in the following table 2. The melting index by the **** company considers as the resin of 10, and the polypropylene resin to be used is NRII made from Japanese MARCROSS. 46mm 2 shaft EKUSUTO loader blocks formula extruder C/D 40 was used and the **-like white polypropylene refractory-material sample was manufactured. The subjects of an experiment at each time contain 10kg of polypropylene resin. Five samples were taken and examined from the latter-part product into before the already extruded object, respectively. Sample specification was set to 0.2cmx1.0cmx15cm, and performed the

combustion test and the surface gloss examination.

押し出し条件:

押し出し温度 200℃

スクリュー速度 50rpm

入口温度

75℃

出口温度

190℃

[Table 2]

376 144 -	•-						
サンフル 番号	利ブロビレン 重量比	TBBPA -DBP	本発明	Sb.O.	臭素 含有量	UL-94 レベル	程現象
1	82	12		6	8. 04	V-0	2 週間後表面白化
2	85		10	5	7. 08	V-2	2 カ月後表面光沢
2	83. 5		11	5. 5	7. 79	V-2	
2	82		12	6	8. 50	V-0	
2	80. 5		13	6. 5	9, 20	V-0	
2	83. 5	11		5. 5	7. 37	V-2	2 週間後表面白化

注: 1.本試験の火炎はライターを使用し、火炎の高さ3cm 、燃焼点は火炎底部より1cm テスト時間は25秒

2. 霜現象は室温、室内空気中で観察する

[0009] this invention is made a low ratio by experiment from 80%, and it is Sb 2O3 of optimum dose. It is UL-94 in the situation distributed uniformly after mixed kneading. The fireproof demand of V-0 can be attained, and even if it compares the fire-resistant effect with other flame retarders, it turns out that it is very good. Moreover, for a user, it is a big merit, and a distributed assistant can be added, it can make with fire-resistant ****, a dispersion effect can be assisted, it is more convenient in use, and it is also economical that it is hard to generate a frost phenomenon. [0010] Various kinds of names and structures of a constituent are explained below.

1. The structure of a hexa BUROMOSAI clo dodecane (Hexabromocyclododecane;HBCD) is as the following chemical structure formulas 1.

2. The structure of deca BUROMO diphenyl (Decabromobisphenyl) is as the following chemical structure formulas 2. [Formula 2]

3. The structure of an octave ROMOJI phenyl (Octabromodiphenyl) is as the following chemical structure formulas 3. [Formula 3]

$$Br_{x} \qquad br_{y} \qquad x+y=8$$

4. The structure of tetrabromobisphenol A (Tetrabromobisphenol A) is as the following chemical structure formulas 4. [Formula 4]

5. The structure of a tetrabromobisphenol A-screw (2, 3-dibromopropyl ether) is as the following chemical structure formulas 5.

[Formula 5]

6. The structure of the screw (3, 5-dibromo -4 (2, 3-dibromo pro PUKISHI) phenyl) sulfone (Bis(3, 5-dibromopropxy) phenyl) sulfone is as the following chemical structure formulas 6. [Formula 6]

[0011] this invention is UL-94. According to [are the flame retarder research and development in was done to V-0 fire-retardancy polypropylene resin, and the fire-resistant effect over polypropylene resin is very remarkable, and] the test result An again very good fire-resistant effect is attained also to shock-resistant polystyrene resin, and it is Sb 2O3 1/3 to addition 5phr by examination. Under the situation of having blended, it is UL-94 to shock-resistant polystyrene resin. The fire-resistant demand of V-2 was able to be made to attain.

[0012]

[Effect of the Invention] When the experiment to the constituent of this invention was compared with various kinds of other effective flame retarders, this invention was effective, and it turns out that it is a flame-retarder constituent with economic effects, and it was illuminated by either the test method of a laboratory, or the actual machine operation method that it is a useful flame-retarder constituent. the constituent of this invention, and FG of the Teijin chemistry Fire of 3100 or **** chemistry Cut Great of 680G or the U.S. Although each fire-resistant effect was good when LakePe68 was compared, this invention is more excellent in the point of being hard to generate economical efficiency and frost, and may coincide with the demand which is a user more.